

AMENDED SPECIFICATION, SHOWING CHANGES TO THE SPECIFICATION

IN RESPONSE TO OFFICE ACTION DATED May 31, 2005

FOR SERIAL NO.(10/706,477) FILED NOVEMBER 7, 2003

Paragraph 15 beginning on page 8:

**[0015]** Microvalves are characterized by their geometry, actuation mechanism, whether they are active or passive, normally open or closed, and whether they contain external or integrated actuators as depicted in ~~Figure~~ Figure 1. Additionally, their membrane material may also be used to characterize them. Membrane materials are typically divided into silicon-based and non silicon-based. ``Normally open"("closed") simply means that the valve is open (closed) when no power is applied. Most MEMS microvalves are silicon-based since they are created using existing silicon chip fabrication methods established by the electronics industry. Newer MEMS fabrication techniques (Lithographie Galvanoformung Abformung (LIGA), LIGA-like, Deep Reactive Ion Etching (DRIE), etc.) permit mass fabrication, including non-silicon parts, with high aspect ratios. Other materials previously incorporated into microvalve designs include Titanium, Nickel, Copper, Aluminum, and silicone.

Paragraph 19 beginning on page 11:

**[0019]** The fuel cell must use a normally open microvalve so that the fuel cell will still operate if the microvalve fails and also to easily permit startup of the fuel cell. The variable operation temperature precludes using the three commercially available valves, which are thermally activated. The four thermally-insensitive actuation mechanisms (and their disadvantages) are:

pneumatic (requires an external air source), piezoelectric stack (low displacement), electrostatic (weak), and electromagnetic (requires external magnetic field). The low displacement of the piezoelectric transducer can be ameliorated by using mechanical structures to amplify their motion (e.g., a trimorph structure). The maximum flow rate was determined by NETL and was based upon the maximum power output of the fuel cell test bed. The desire to operate off of the cell voltage would eliminate the need for an external power bus to the embedded microvalves. It is further hoped that proportional control based upon feeding back the cell voltage would be effective for flow management.

Paragraph 29, beginning of page 14:

**[0029]** Further, the deflectable member may include an intermediate layer interposed between the first and second piezoelectric portions. The deflectable member may include an intermediate layer that is formed from brass or another ductile material. The valve body may further include a cavity formed therein, at least a portion of the deflectable member being disposed in the cavity. The deflectable member of the microvalve may include a fixed end and a free end, the fixed end being secured to the valve body; and the deflectable member including a gate disposed at the free end, at least a portion of the gate being receivable in the flow channel to resist the flow of the fluid through the flow channel when the deflectable member is in the closed position.

Beginning with paragraph 38 starting at the bottom of page 15

**[0038]** ~~Fig. 3e shows a schematic of the microvalve in the open and closed position;~~

[0039] Fig. 3f ~~3e~~ shows the stacking of the microvalves;

[0040] Fig. 4 ~~is a computer simulation of the deflection of PZT trimorph actuator for a 10-volt input~~ is a computer graphic of the Von Misses stresses on actuator resulting for the 10-volt input;

[0041] Fig. 5 ~~is a computer graphic of the Von Misses stresses on actuator resulting for the 10-volt input~~ is a computer simulation of the deflection of PZT trimorph actuator for a 10-volt input; and

Paragraphs 44 and beginning on page 16:

[0044] The microvalve of this invention is shown generally in Figure 3 3a at **10** in spaced apart fashion. Figure 3b illustrates the microvalve as an assembled unit. The microvalve comprises an upper member part **12**, a middle member wafer part **14**, an actuator **16** and a lower member part **20**. Flow is generally from left to right through the microvalve. At the heart of the valve is a piezoelectric trimorph actuator **16** that is used to open and shut an axial flow valve via a valve gate **18**. The valve gate can be formed from materials such as, but not limited to stainless steel or silicon. Three wafers **12, 14, and 16** are fabricated out of silicon to create the flow channel and support the actuation mechanism. The middle member part **14** or wafer serves to shield the actuator **16** from the fluid pressure forces. The gate **18** moves through port **22**. Figure 3b shows a dimensioned drawing of the side view of an assembled microvalve. Note that the piezoelectric actuator **16** mechanism extends past the valve body on the ~~left hand~~ left-hand side, permitting the electrical connections to be easily made. The microvalve is shown in the normal open position in Figure 3c. When a voltage is applied the gate moves through port **22** to block the flow as shown in Figure 3d. ~~Figure 3e shows a simplified view of the microvalve~~

~~moving from the opened to the closed position.~~ The units can be readily stacked as shown in ~~Figure 3f~~ Figure 3e. The microvalves can be installed in the cells of a fuel cell such as a PEM.

There are many novel attributes about this valve design, including:

1. Scalable geometry in height (by stacking) and width
2. Axial Flow
3. Relatively Simple
4. Non-thermally activated
5. Low-voltage operation
6. Linear actuator response
7. Possibly linear flow characteristic

#### **Piezoelectric Trimorph Actuation Mechanism**

**[0045]** A trimorph actuator is created by sandwiching a 25 $\mu$ m thick brass shim between two 127- $\mu$ m thick lead zirconate-titanate (PZT-5H) patches. Parallel electrical connections are made between the patches, which are oriented to have parallel polarization directions. This configuration provides ~~the most~~ higher deflection for a given voltage compared to previous valve designs based upon a piezoelectric stack.

Paragraphs 49 and 50 beginning on page 49:

**[0049]** A coupled thermal-electrical-mechanical analysis of the actuation mechanism for the valve was performed using ANSYS ~~cite~~ and MATLAB ~~cite~~ ~~models~~. In addition, the flow through the microvalve was analyzed using the same software packages. Finally, the thermal effects were added to the analysis. The next sections treat the electro-mechanical, flow, and thermal analyses, respectively. These models were used to perform a parametric study to optimize the geometry, forces, flow, and fabrication of the valve.

#### **Electromechanical Analysis**

**[0050]** The final actuator mechanism design has dimensions of 22500 x 4000 x 290 microns. The thickness of 290  $\mu\text{m}$  includes two PZT layers of 127 $\mu\text{m}$  each, a 25- $\mu\text{m}$  brass layer and two glue layers that should not exceed 11 $\mu\text{m}$ . ANSYS coupled field element 5 and brick element 45 were used to model the piezoceramic layers and the inner brass layer, respectively. A mesh convergence was performed and a mesh size of 200 microns was used. Both deflection and stresses were analyzed for a 10-volt (the maximum permitted for this application) excitation on the piezoceramic. The deflection is shown in ~~Figure 4~~ Figure 4 and 5 and was used to determine the maximum height of the valve flow channel, when considering the fluidic forces. A maximum deflection of 64  $\mu\text{m}$  is obtained for the 10-volt excitation. The von Mises stresses are shown in Figure 4 and were used to determine if the materials would fail during actuation. The maximum observed stress is 6.4 MPa. Since the failure strengths of PZT and brass are 63 and 270 MPa, respectively, the factor of safety for the device is about 10 in the absence of fluid and thermal loads. Note that the final valve design calls for only a 5-volt excitation since only 30 microns of deflection is required. The stresses will subsequently decrease by half as much for the 5-volt excitation as well (3.2 MPa).

Paragraph 52 beginning on page 20:

**[0052]** Finally, the force deflection diagram for the piezoelectric actuator can be found by connecting the straight line between the blocked force (zero deflection) and free deflection (zero force) conditions. A free deflection of 32 microns at 5 volts has been selected for the operating point. The blocked actuator tip force (that is the tip force required to prevent deflection when a voltage is applied) of  $F_B = 7.3 \text{ mN}$  for a 5-volt excitation was determined iteratively using ANSYS. ~~Figure 7 shows the resulting force-deflection diagram for the actuator.~~ The slope of this line can be determined from the two end points as  $m = (7.3\text{mN})/(-32\mu\text{m}) = -228 \mu\text{N}/\mu$   ~~$(7.3\text{mN})/(-32\mu\text{m}) = -228 \mu\text{N}/\mu$~~ . Note that a 5-volt excitation is only 8% of the 0.5 Mv/m depolarization field for the piezoceramic.

Paragraphs 58, 59 and 60 beginning on page 22:

**[0058]** The pressure drop across the valve gate will create an additional horizontal force, where  $\Delta P$  is the pressure drop across the gate and  $A$  is the exposed area. For the maximum pressure difference of  $\Delta P = 1 \text{ psig}$  and the maximum exposed area of  $A = 12 \times 10^{-8} \text{ m}^2$  (valve closed) the pressure force is calculated as  $F_p = 830 \mu\text{N}$ . The resulting moment and vertical bender tip force are:  $M_{p,eq} = 0.28 \mu\text{N}\cdot\text{m}$  and  $F_{p,eq} = M_{p,eq}/d = 14 \mu\text{N}$ , respectively. The total vertical force on the bender due to fluid effects is the sum of the two contributions =  $30 \mu\text{N}$ . In ~~Figure 8, the linear force-deflection diagram for the piezoelectric bender was presented along with the blocked force of 7.3 mN, the free deflection of 32  $\mu\text{m}$ , and the slope of -228  $\mu\text{N}/\mu$ . The resulting loss in deflection due to the fluidic force resultant of 30  $\mu\text{N}$  can be determined from the slope of the force-deflection diagram (Figure 8) as  $x_{loss} = 30 \mu\text{N} / 228 = 0.13$~~

$\mu\text{m}$ , which is less than 0.5% of the free deflection of  $32\ \mu\text{m}$ . Note that the pressure port shown in ~~Figure 3~~ Figure 3a serves to equalize the pressure across the piezoelectric trimorph actuator, preventing undue additional forces. ~~Table 3 summarizes the fluidic force calculations for the valve.~~

### Thermal Analysis

[0059] Finally, the thermal effects were added to the analysis to ensure that thermally induced stresses will not cause the valve actuator to fail and to ensure that internal clearances are sufficient given the nominal operating temperature of ~~100°C~~ 100°C. The ANSYS model was used to perform the coupled electrical, thermal, and mechanical analysis. Since the valve will be cured at ~~100°C~~ 100°C, and cooled to room temperature of approximately ~~20°C~~ 20°C, a temperature differential of ~~80°C~~ 80°C was considered. ~~Table 4 summarizes the forces, voltage and thermal loads on the actuator. The thermal properties that were used for the brass and piezo layers are given in Table 5.~~ Thermal conductivity of the lead zirconate titanate PZT-5H is estimated by dividing Aluminum's conductivity by 200 (personal communication, Piezo Systems, Inc.)

[0060] ~~Figure \ref{fig:thermal\_long} shows the elongation of the actuator. It can be seen that the bender elongates  $6\ \mu\text{m}$ , and thus a minimum clearance of  $10\ \mu\text{m}$  is chosen to prevent binding.~~ ~~Figure \ref{fig:thermal\_misses}~~ Figure 5 shows the von Mises stress distribution over the actuator for the fully coupled analysis. The stress level on most of the parts is between 2 MPa and 9 MPa. A maximum stress of 63 MPa occurs on the brass shim near the cantilevered edge. The maximum stress on the piezo layers is found to be around 40 MPa, which occurs on the cantilevered end and around the gate. Since the strength values for piezo and brass are 63 MPa and 270 MPa, the microvalve will operate safely under maximum temperature difference. Note also, that the maximum thermal stress occurs at room temperature (when not in service)

since the actuator is cured at elevated temperature and then cooled. ~~Therefore~~ Therefore, the operational factor of safety will actually be much higher than  $63\text{MPa}/40\text{Mpa} = 1.5$ . The resulting residual thermal stresses that occur from cooling will disappear as the valve is heated to operating temperature, which is the primary mode of operation for this valve.

Paragraph 64 on page 24

### **Micromachining of Silicon Valve Body**

[0064] The valve body is constructed from single crystal silicon substrate and requires high-aspect ratio machining. ~~Much~~ Such machining is best performed using LIGA (Lithographie Galvanoformung Abformung), LIGA-like, or deep reactive ion etching (DRIE). The latter will be used in this study. DRIE etches using a plasma stream that can be tailored to different materials by selecting the appropriate ionized gas. It is a subtractive process, whereby a mask is created on the surface of the stock, which will shield the parts that are not to be machined. The depth of the machining will be controlled by the length of time that the masked device is exposed to the etchant. For devices that have grooves or holes machined down to different levels, e.g. the lower and middle wafers, multiple masks and subsequent DRIE cycles ~~will be~~ are required. After machining, the etch resist is chemically removed and the parts cleaned.



Paragraph 66 on page 25:

[0066] A piezoelectric microvalve has been designed and analyzed. The valve is to operate inside the hydrogen flow stream of the PEM fuel cell stack used for the flow control energy management research at NETL-Morgantown. Materials have been selected which have good hydrogen resistance. ANSYS was used to analyze the coupled electrical, thermal, and mechanical response. A fluidic analysis was also performed to determine the flow characteristics as well as the actuator forces that result from the fluid motion and pressure drop. It was found that the valve could withstand the forces resulting from thermal, fluid, and piezoelectric loading. Finally, a fabrication plan has been developed to create the valve. ~~Table 6~~ Table 1 summarizes the final geometry, operating conditions and results such as maximum deflection, and maximum stress values over the actuator. It also gives the failure strengths for actuator materials for a quick comparison with maximum stresses on them.

Paragraph 71 on page 27:

[0071] ~~An initial design approach used the brass shim as a common electrode, and the electrodes on the inner sides of each plate were etched with ferric chloride. Since the glue layer is very thin, the brass layer was used as a common electrode. For actuation, the piezo ceramic layers are oriented to have parallel polarization directions. Figure 2 shows the polarization orientation of the piezo ceramics. It was found that the actuator had a loss of capacitance in piezo layers, accompanied by degraded performance. Thus a second fabrication method was used where no etching of the PZT electrodes is performed, which requires an additional electrical connection.~~

Paragraph 73 on page 28:

**[0073]** An improved way to apply pressure during the cure process is to use spring clamps. Silicone gum pads and aluminum back plates are used together with the clamp to hold the actuator. The area and clamping spring rates were chosen to give the optimal 30-35 psi pressure. The inner parts of the silicone pads are covered with Teflon films to prevent sticking of the actuator to the pads while the actuator is being cured. This method is observed to overcome the problems associated with the vacuum system. The actuators that are fabricated using the spring clamps are 290 $\mu$ m thick which is the design thickness value. They are also free of any non-uniformity in thickness. The spring clamp setup is cheap and easy to build. It also eliminates the need for electricity for the vacuum process and decreases the cost. ~~Figure 3 shows the clamping equipment and setup used for curing.~~

Paragraph 75 and 76 on page 29:

**[0075]** Once the actuators are cut, they are ready for the final step of the fabrication, which is wiring. ~~For the etched actuators, the brass shim can be used as a common electrode. This configuration needs three electrical connections. If the brass shim weren't used as a common electrode, then four~~ Four connections are required (one for each PZT electrode), which are achieved by soldering. ~~This requirement complicates the fabrication since only one side of each beam is accessible. Two different methods were evaluated for wiring. First, a conductive epoxy was tried to connect the wires to the actuators. This epoxy needs four hours of curing. All electrical connections cannot be made at the same time since one of them is on one side of the actuator and the other two are on the opposite side. Wiring two sides separately, it takes more~~

~~than eight hours to establish the connections. To decrease the fabrication time, soldering was used for the later actuators instead of gluing. It decreases the required time for the whole wiring process to 10 minutes, and has little affect the actuator performance. Detailed information about the performance of the actuators that are fabricated using different techniques is given in the next section.~~

**[0076]** Once the actuator fabrication is complete, a silicon valve gate is added to the end. The valve gate has dimensions of ~~200×230×4000μ~~ 200×230×4000μm. It is first diced from a silicon wafer to the proper dimensions, and then it is bonded to the actuator using the same epoxy as used in the other fabrication steps. A Teflon jig is used support the gate during cure to ensure that it is mounted at a right angle to the actuator.

Paragraph 79 on page 30:

**[0079]** Each of the three silicon valve parts is fabricated on separate silicon substrates. Six valve parts were laid out on each silicon wafer. ~~Figure 5 shows the bottom layer of the microvalve fabricated on a silicon wafer. Only four of the six wafers are shown in this figure.~~

Paragraph 84 on page 31:

**[0084]** Once the calibration curve is obtained, the probe is set at a distance from the test surface near the midpoint of the linear region of the front slope of the calibration curve. Then, a controlled input voltage is applied to the actuator and the change in the probe output voltage is recorded. Using the change in the output voltage and the calibration curve, the corresponding deflection value is calculated for the applied input voltage. Deflection tests were repeated for

different input voltages to verify the tip deflection and voltage-deflection relation. ~~Figure 7 shows the photonic probe setup and the actuator during the test.~~

Paragraphs 86 and 87 on page 32:

[0086] From Table 2, it can be seen that the average deflection of the actuators is 30.3  $\mu\text{m}$  when an input voltage of 5 V is applied. This is only 1.67  $\mu\text{m}$  or 5.2% less than the theoretical deflection value obtained from the ANSYS model. This much loss in deflection is acceptable for this application.

**~~Figure 7~~ Deflection test setup**

[0087] After the room temperature tests, actuators are tested at elevated temperatures up to 100°C. The test probe and the fixture are placed in an oven and heated up to 100°C. Without applying any voltage the output voltage is recorded to determine any thermal deflections. There are two important issues for the high temperature tests, the thermal drift of the probe, and a permanent darkening of the brass, which changes the reflectivity of the material. To determine the thermal drift of the probe, the probe was installed on a solid brass surface and heated. This process was repeated several times and it was verified that the probe has a thermal drift of -0.24 V for a temperature difference of 80°C on brass surface. To eliminate the brass darkening effects, the brass shim is heated and kept at 100°C for approximately 20 minutes before the tests. Then the probe is calibrated on the darkened brass. For one actuator, at least four high temperature tests per side were conducted. Although the first actuators deflected thermally, after the refinements in fabrication process (clamping), actuators without any thermal deflection have been built. The second phase of the high temperature tests is to determine the actuation deflection at high temperatures when a voltage is applied. An input voltage of 5V is

applied to the actuators while the ambient temperature is kept at 100°C and it is observed that the actuators function safely and effectively at high temperatures. Deflection values obtained in these tests are exactly the same with the numbers obtained in room temperature tests.

Paragraph 92 on page 34:

**[0092]** With this description of the invention ~~in~~ in detail, those skilled in the art will appreciate that modification may be made to the invention without departing from the spirit thereof. Therefore, it is not intended that the scope of the invention be limited to the specific embodiments that have been illustrated and described. Rather, it is intended that the scope to the invention be determined by the scope of the appended claims.